



Characterization of Zeolite as an Adsorbent for Capturing CO₂

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The characterization of zeolites as an adsorbent for capturing CO₂ from biogas has been done chemically and physically. The chemical treatment is using H₂SO₄ as an activator with varying concentrations in the range of 5%–30% and physical treatment is calcination at 500 °C for 4 hr. Adsorption process carried out by flowing CO₂ synthetic gas (98.86%) from the bottom of the adsorber column with a flow rate of 0.22 cc/min until 0.58 cc/min. Adsorber column have a diameter 5 cm and height of 30 cm, height of adsorbent 28 cm and the grain size of the zeolite –20/+28 mesh. The research resulted in the best condition in chemical activation of 25% H₂SO₄ and calcination increasing ratio Si/Al of 5.5 to 9.73. Characterization of zeolites have a total pore volume of 15.001 cc/g, pore surface area of 998.032 m²/g, mean pore radius 28.997 Å and CO₂ absorption capacity of about 75.5% at the gas rate penetrating to the adsorber of 0.22 cc/minute.

Keywords: Zeolite, Adsorbant, Calcination.

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1. INTRODUCTION

Waste liquid vinasse from alcohol industry have been processed anaerobically using activated sludge to produced biogas. The waste water treatment of Vinasse from the fermentation industry of anaerob alcohol taken from the activated sludge of cow dung treatment can produce biogas containing methane: (CH₄) 42,89–58,06%, and carbon dioxide: 41,94%–58,11%.¹ The gas content of CH₄ more than 60% can be obtained by removing CO₂ gas using some adsorbent. Adsorbent is a material which is capable of adsorbing the gas based on its porous characteristics. This porous material was chosen because its inside surface was wider than the outside one.²

The use of porous material like sepiolite, silica and zeolite as gas adsorbent has been very popular. The combination of Fe₂O₃ and Fe₃O₄ with monmorillonite to adsorb H₂S gas contained in biogas had been conducted.³

Meanwhile, used zeolit from faujasit of, NaX and NaY as an adsorbent to remove H₂S gas contained in biogas.⁴ In order to get rid of CO₂ gas from the mixture of CH₄ gas and CO₂, we used sepiolite through pressure swing adsorption.⁵ Based on the former research, it was stated that zeolit was capable of adsorbing the gas.

Zeolit is a compound of alumino hydrated silicate containing of the bond of hydrated SiO₄ and AlO₄ connected by some oxygen atom to form a framework. Furthermore, in zeolite framework, each Al atom has a negative properties. This condition can

be neutralized by using the exchangeable cation. These cations which are easily to be exchanged are available in the zeolit framework. This might affect to the adsorption process and its thermal zeolit properties.⁶ Furthermore, the type of cation enabling to adsorb zeolit can also be influenced by the percentage of Si/Al and its geometrical pores of zeolit as well as its inside surface width of pores and its shape.^{7,8} The ratio change of Si/Al from zeolit is capable of changing the zeolit charge, so that finally it can change the number of counterweight cation. Less Al atom means that the amount of zeolite containing negative charge is less so that the counterweight cation contained in zeolite will also be less than that of the high zeolite having hydrophobic properties and having its affinity to hydrocarbon.

In line with the varieties of zeolit structure and its impurities available, it was suggested that before being used, zeolite had to be treated formerly which was called as activation process. In general, activation process to zeolit was conducted physically by heating it for several hours and chemically by using acid or base. Activation process of zeolite conducted chemically by using acid or base could be obtained that the activated zeolite by using acid or base would become more polar than that of the activated zeolite using acid.⁹ The treatment using acid to the zeolit might cause the zeolite become more hydrophob so that its adsorption potency to the water would become less.¹⁰

Furthermore, the adsorption potency of gas was influenced by its adsorbent texture. Gas adsorption required some adsorbent of micro size ($d \ll 20 \text{ Å}$) because gas had a molecule size, that was $< 4 \text{ Å}$.¹¹ Adsorption process of gas by adsorbent was affected by

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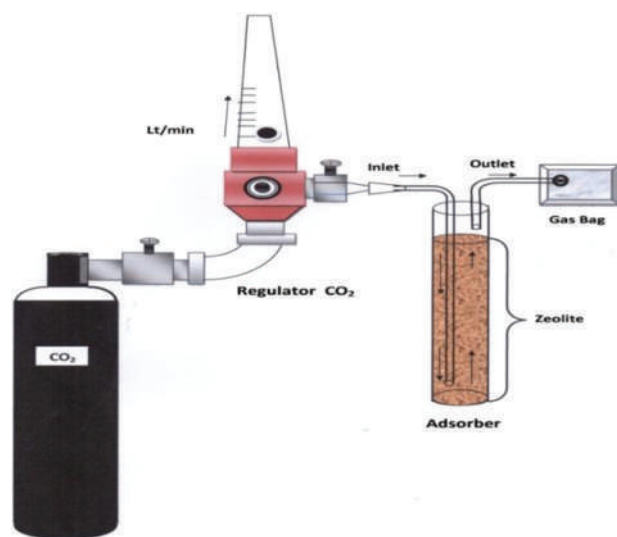


Fig. 1. Diagram of adsorption CO₂ process with zeolite adsorbent.

the interaction of van der Waals related to polarisability of molecular adsorbent and adsorbent. The wider the surface of adsorbent, the more often the interaction of van der Waals will happen. This might make the gas absorption become higher.¹¹ If the molecule size is compared with the size of pore adsorbent, the gas will be adsorbed into its adsorbent. This occasion is called sterical mechanism.¹² Based on this is it necessary to characterize zeolite.

2. METHODOLOGY

CO₂ gas adsorption using zeolite done using equipment such as arranged in Figure 1. The adsorption method is carried out experimentally in three stages. The first stage is the design of the adsorber column having a diameter of 5 cm, height 30 cm high and 28 cm of adsorbent high. The second stage is a zeolite having a grain size of $-20/+28$ mesh chemically activated using sulfuric acid with concentrations varied in the range 5% to 30%

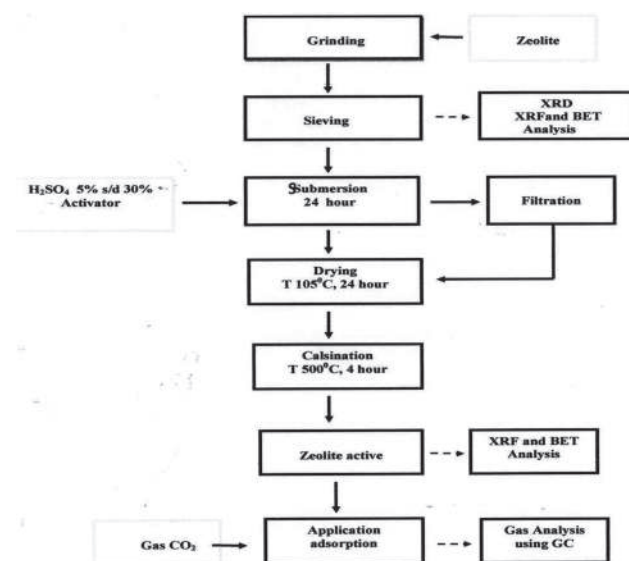


Fig. 2. Diagram of methodology.

while the activation is physically carried out calcination at a temperature of 500 °C for 4 hours. The third stage is the adsorption process conducted by flowing CO₂ gas 98.86% from the bottom of the adsorber column with a flow rate varies in the range of 0.22 cc/min up to 0.58 cc/min. Gas exit adsorbent accommodated in the gas bag for purposes of determining the levels of CO₂.

X-ray diffraction zeolite was done for to determine the constituent mineral zeolite, while the zeolite composition analysis using X-ray Fluorescence. Characterizations of zeolites were analyzed by gas-desorption N₂ adsorption using the equation Brunauer, Emmet and Teller (BET) and gas out of the gas content of the adsorbent analyzed using GC.

3. RESULTS AND DISCUSSION

Figure 3 shows X-ray diffraction (XRD) pattern of particles zeolite, the components of zeolite mostly modernit and quartz.

Figure 4 shows the Si/Al ratio on particle zeolite before and after activation. The Si/Al ratio without activation is 5.5, while the activation of zeolite at a concentration of 25% H₂SO₄ produces Si/Al ratio reached 9.73.

The increasing the ratio of Si/Al resulted in a decrease in pore size due to Si–O chain is shorter than the chain Al–O so that the

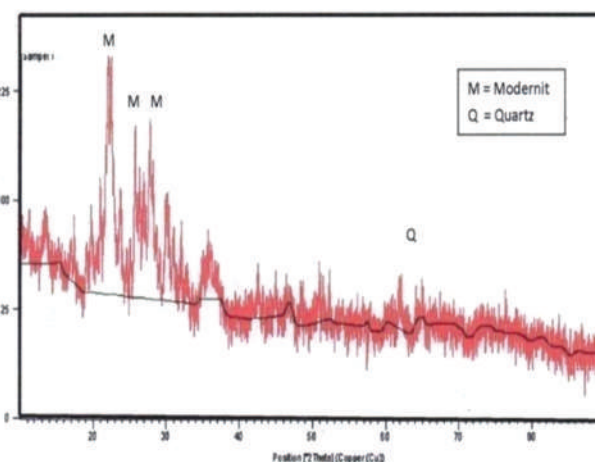


Fig. 3. X ray diffraction of zeolite particle.

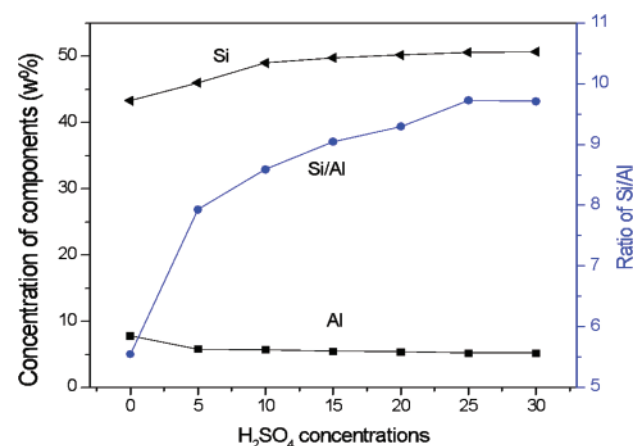


Fig. 4. Si/Al ratio on particle zeolite before and after activation.

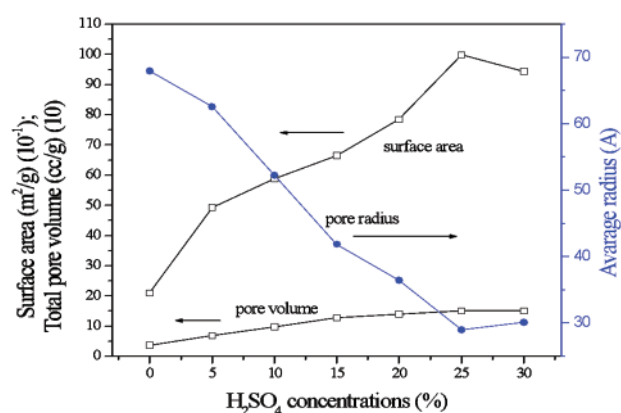


Fig. 5. Effect of H₂SO₄ on surface area, total pore volume and average pore radius of particle zeolite.

frame size is smaller. The decline was also accompanied by an increase in pore surface area thereby increasing contact between the adsorbent and the adsorbate (Fig. 5). Calcination aims to vaporize Bronsted bases and H₂O as well as arranging atoms mixed up to form stable metal oxides.

The surface area (BET) and total pore volume tends to increase with increasing concentration of H₂SO₄ activator, while the average pore radius decreases. The pore size of the adsorbent will affect the absorption of gas. Figure 5 shows the pore size of the

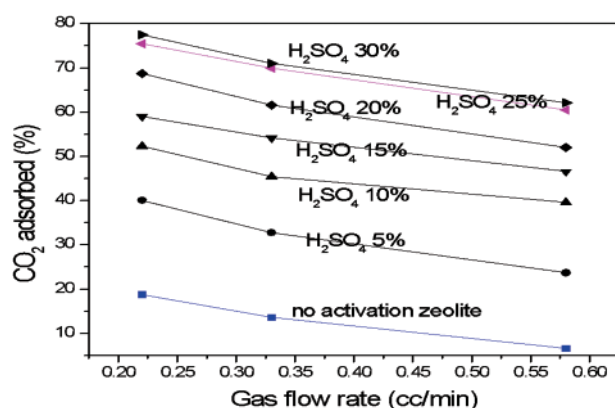


Fig. 6. Effect of gas flow rate on CO₂ adsorbed.

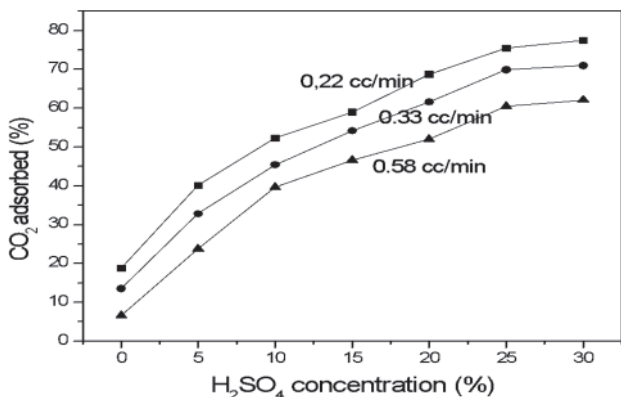


Fig. 7. Effect of H₂SO₄ concentrations on CO₂ adsorbed.

Table I. Components of zeolite (XRF).

Component	Concentration (%)					
	Blanco	H ₂ SO ₄ 5%	H ₂ SO ₄ 10%	H ₂ SO ₄ 15%	H ₂ SO ₄ 20%	H ₂ SO ₄ 25%
Si	43.3	46	49	49.8	50.2	50.6
Al	7.8	5.8	5.7	5.5	5.4	5.2
Ca	11.9	15.9	13.8	15	12.4	13.1
K	12.3	10.4	10.6	10.3	10	9.22
Fe	8.75	7.25	6.7	6.57	6.24	5.07
Na	3.2	2.6	1.8	1.4	0.9	0.7

zeolite ranges between 3.6 cc/g to 15.001 cc/g has the potential to absorb CO₂. CO₂ flow rate effect on the adsorption process. Figure 6 describes the ability of the zeolite to adsorb CO₂. Percentage of CO₂ gas adsorbed is increases as the size of the flow rate of CO₂ that enters the adsorber column. Differences on the increase in the percentage of CO₂ adsorbed are ranges from 5–11% for every decrease in the rate of CO₂ entry in the range of 0.22 cc/min to 0.58 cc/min. The percentage of CO₂ gas was adsorbed increased with increasing concentrations of H₂SO₄ activator. H₂SO₄ dissolves impurities that cover the pores, so that the zeolite pores to be open and the surface becomes more widespread. Figure 7 shows the percentage of CO₂ gas is adsorbed into the zeolite increased with increasing concentrations of H₂SO₄ activator further decreased at the rate of 30% activator. This is because the concentrated acid that causes the large number of Al-soluble so many atoms out of the zeolite framework structure of the zeolite causing damage. As a result, adsorption ability decreases.

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4. CONCLUSIONS

Activated zeolite prepare by sulfuric acid as activator have done successfully. The percentage of CO₂ adsorbed dependent to the concentrations of H₂SO₄. The activation of zeolite using sulfuric acid can affect the value of ratio Si/Al. A good result of it could be obtained well if zeolite chemically treatment was conducted by using H₂SO₄ activator 25% and physically treatment is calcinations at 500 °C for 4 hr which could increasing ratio Si/Al of 5.5 to 9.73. Characterization of zeolites have a total pore volume of 15.001 cc/g, pore surface area of 998.032 m²/g, mean pore radius 28.997 Å and CO₂ absorption capacity of about 75.5% at the gas rate penetrating to the adsorber of 0.22 cc/minute.

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